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(54) **Dry Blend Preparation**

(57) A method of preparing a polyvinyl chloride dry blend from solid vinyl chloride polymer dry blend grade resin particles, solid filler particles and

liquid plasticizer in which separation of filler particles during processing is reduced by adding together with the plasticizer at least 0.2 part per 100 parts by weight of resin of a liquid cationic, nonionic or amphoteric surfactant.

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SPECIFICATION

Dry Blend Preparation

This invention relates to thermoplastic sheets suitable for use as floor coverings, dry blends for use in their manufacture, and a method of preparing the dry blend.

5 More especially, this invention relates to floor coverings produced from a vinyl chloride resin dry blend. 5

In one of its more specific aspects, this invention relates to a vinyl chloride resin dry blend formulation which exhibits excellent processability.

10 Vinyl resin dry blending is a well known compounding technique used in preparing polyvinyl chloride formulations for extrusion or sheet layup. Dry blending involves mixing the solid constituents, that is, polyvinyl chloride dry blend grade resin, filler, and, if desired, a lubricant and a solid stabilizer, followed by adding a liquid plasticizer and heating the mixture to a temperature above the "fluff temperature" of the resin particles so that a dry blend, i.e., a dry, free flowing, sandy powder of uniform composition, results. If liquid lubricants or liquid stabilizers are employed, they are normally premixed with the plasticizer. Typically, to produce a floor covering, the finished powder mixture is laid up on a release carrier, sintered to form a porous layer, printed using vinyl plastisol inks, fused and consolidated under pressure to form a decorative floor covering. 15

Certain processing difficulties are associated with the manufacture of filled vinyl resin dry blends. One is that during processing the filler particles tend to separate from the dry blend formulation and build up on the layup or screed roll. 20

The present invention helps solve these processing problems and provides a filled vinyl resin dry blend formulation which facilitates processing by eliminating or reducing filler separation and resin and filler buildup on layup or screed rolls, and by requiring less oven dwell time to achieve optimum sintering. Also, the resulting surface, usually floor, coverings exhibit glossier, smoother surfaces than the prior art coverings. 25

According to this invention, there is provided a method of preparing a vinyl chloride polymer dry blend from solid vinyl chloride polymer dry blend grade resin particles, solid filler particles and liquid plasticizer wherein the solid particles are blended, followed by the addition of the liquid plasticizer with heating to at least the fluff temperature of the resin particles such that the liquid plasticizer is uniformly and rapidly absorbed through to the core of each resin particle producing a dry powder of uniform composition, wherein there is mixed and added together with the plasticizer at least 0.2 part per 100 parts by weight of resin of a liquid cationic, nonionic or amphoteric type hydrocarbon-, silicone- or fluorocarbon-containing surfactant. 30

The invention further provides the dry blend prepared by said method.

35 The invention also provides a dry blend comprising plasticized vinyl chloride resin particles, and filler particles, the composition comprising at least 0.2 per cent by weight, based on the weight of the unplasticized resin, of a cationic, nonionic or amphoteric hydrocarbon-, fluorocarbon or silicone-containing surfactant. 35

The present invention further provides a decorative surface covering, especially a floor covering produced by screeding a dry blend of the invention to form a uniform gauge layup, sintering the layup to form a porous sheet, printing the porous sheet with vinyl plastisol ink, and simultaneously fusing and consolidating the printed sheet to produce the covering. 40

As used herein, the term "fluff temperature" is that temperature, typically between 130°—195°F. (about 55°C to 90°C), at which the particles of dry blend resin start to take in plasticizer by absorption and become solvated. 45

If the surfactant is a silicone-containing surfactant, it is preferably an organo-modified silicone surfactant.

If the surfactant is a hydrocarbon-containing surfactant, it is preferably an alkylaryl polyether alcohol surfactant. 50

As the base vinyl dry blend grade resin, use can be made of any vinyl homopolymer or copolymer resins conventionally employed in dry blend compounding. 50

A dry blend grade resin is one that will uniformly and rapidly absorb all the plasticizer employed in the formulation. This does not mean merely drying up the blend, but means that each resin particle is plasticized through to the core, with a substantially uniform concentration of plasticizer. Resins which do this best are those which act like a sponge with respect to plasticizer, for example "Opalon" (registered Trade Mark) 660, 650 and 630, representative of Monsanto's "600" series of polyvinyl chloride dry blend grade resins, and "Exon" 9290, a dry blend resin commercially available from Firestone. 55

A true dry blending grade resin should not be confused with a resin which will merely premix to a dry state. The latter type makes use primarily of its surface area to absorb plasticizer, whereas a true dry blending resin takes in plasticizer by absorption and becomes solvated. In the case of the true dry blending resin, each particle is capable of taking up its proportionate share of plasticizer, and this minimizes formation of "fisheyes," or unplasticized particles, which show up in the surface of a finished sheet. 60

As will be apparent from the discussion below, the composition may contain in addition to the essential dry blend vinyl resin other vinyl resins, e.g. blending grade vinyl resins.

As the plasticizer, use may be made of any general type plasticizer or plasticizer system, many of which are conventionally employed in dry blend compounding.

5 A high solvating plasticizer is preferred since it tends to give the most complete solvation in the 5 fastest time. However, both monomeric or ester type plasticizers and polymeric plasticizers may be used as well as low cost extenders and mixtures thereof. Particularly suitable plasticizers for use herein are dioctyl phthalate and butyl benzyl phthalate.

10 From 20 to 130 parts of plasticizer will normally be employed per 100 parts by weight of resin. 10 The dry blend formulation will advantageously contain at least one stabilizer, preferably being employed in a total amount of from 1 to 10 parts per 100 parts by weight of resin.

Especially suitable stabilizers include epoxidized soya bean oil, barium neodecanoate, zinc neodecanoate, organo-tin stabilizers, and mixtures thereof.

15 The dry blend formulation will preferably contain from 1 to 300 parts of filler per 100 parts by 15 weight of resin.

Any suitable filler particles can be employed. Particularly suitable fillers are alumina trihydrate and limestone.

20 A particularly suitable filler is that designated "KH31F" alumina trihydrate commercially available 20 from Kaiser Chemicals. KH31F has a particle distribution as follows: 4—10% on a 100 mesh screen, 50—70% on a 200 mesh screen, 85—95% on a 325 mesh screen and 3—15% through a 325 mesh screen; the sizes given being U.S. mesh sizes.

25 If the average particle size of the filler employed is 100 mesh or finer or if more than 50 parts of 25 filler per 100 parts of resin are employed, it has been found that the substitution of up to 20 percent (20 parts) of a conventional vinyl chloride homopolymer or copolymer blending grade resin for dry blend resin serves to provide better adhesion between the filler particles and the dry blend resin 25 particles.

The dry blend formulation contains at least 0.2 parts, and, preferably, from 0.2 to 2 parts, of the liquid surfactant per 100 parts by weight of resin.

30 Although the correlation between surfactant performance and chemical composition of the dry 30 blend, a correlation known *per se*, is sufficient for the selection of a suitable surfactant, in many cases the selection of the best surfactant for a particular dry blend formulation is empirical, based upon trial and error.

35 It has been found that surfactants having groups selected from each of the three major chemical 35 classes, hydrocarbons, silicones and fluorocarbons, can be employed, and that these surfactants may be cationic, nonionic or amphoteric. Anionic surfactants have been found not to improve processability.

40 It has also been determined that the amount of surfactant employed may be as little as 0.2 part 40 and, that more than 2 parts of surfactant do not appreciably improve processability. In evaluating a particular surfactant for use, it has been found that 1 part of surfactant per 100 parts of dry blend resin is generally a suitable starting amount which can then be empirically increased or decreased to obtain optimum processability.

One particularly suitable silicone surfactant is commercially available as Union Carbide L-7607 from Union Carbide Corp. L-7607 is an organo-modified silicone surfactant.

45 One particularly suitable hydrocarbon surfactant is commercially available as "Triton" (registered 45 Trade Mark) X-100 from Rohm & Haas Co. "Triton" X-100 is a liquid, nonionic octylphenoxy polyethoxy ethanol surfactant having an HLB of 13.5.

Particularly suitable fluorocarbon surfactants include "Fluorad" (registered Trade Mark) FC430, a nonionic, fluorinated alkyl ester surfactant commercially available from 3M Co. Chemical Division and Du Pont "Zonyl" (registered Trade Mark) FSN and "Zonyl" FSC a nonionic fluorochemical surfactant and cationic fluorochemical surfactant, respectively, both commercially available from Du Pont.

50 The following examples, of which Example 2 is a comparison example, illustrate the invention. All 50 parts are parts per 100 parts by weight of resin.

Example 1

55 Into a premix tank with mixing were introduced the following amounts of liquid constituents: 40 55 parts of dioctyl phthalate, 5 parts of epoxidized soya bean oil, 1 part of octylphenoxy polyethoxy ethanol surfactant ("Triton" X-100), 1 part of zinc neodecanoate and 1 part of barium neodecanoate.

Into a high shear blender were introduced the following amounts of solid constituents: 90 parts of polyvinyl chloride dry blend grade resin ("Exon" 9290), 10 parts of polyvinyl chloride blending grade resin ("Plivoc" (registered Trade Mark) M50-Goodyear Chemicals), 2 parts of zinc oxide pigment, and 50 parts of alumina trihydrate particle filler ("KH31F") Kaiser.

60 The solid constituents were mixed and the contents of the premix tank were added to the 60 contents of the blender with mixing and heating to a temperature of about 225°F. (about 107°C) which is above the 185°F. (85°C) fluff temperature of the resin particles. Mixing was stopped when a powder of uniform composition formed.

The resulting composition was cooled to room temperature to provide the dry blend formulation of this invention. The dry blend was sifted through a 30 mesh screen to remove large particles and make the dry blend more uniform for screeding. The dry blend was then screeded to a uniform gauge layup of about 75 mils (about 1.9 mm) on a fibrous release carrier using a textured screeding roll.

5 The dry blend layup was then sintered, that is, the dry blend resin particles were heated to a temperature of about 375°F (about 190°C) for about 2 minutes, which sufficed to cause the particles to become tacky and adhere to each other without noticeably affecting the particle shapes. A porous sheet resulted. 5

10 The resulting sintered porous sheet was then conventionally through-color printed with a vinyl plastisol ink which filled the porous or void areas of the sintered sheet. 10

The printed sintered sheet was then simultaneously fused at a temperature of about 460°F (about 238°C) period of about 1-1/2 minutes and consolidated by the pressure of a laminator into a solid sheet and recovered as a dry blend floor covering having an average gauge of 60 mils (about 1.5 mm).

15 Example 2 15

Using substantially the same procedure and materials of Example 1, with the exception that no surfactant was employed, a second floor covering was produced.

20 During the preparation of both the dry blend floor covering of this invention (Example 1) and the prior art dry blend floor covering (Example 2), the processing properties of each dry blend formulation were observed during sifting, after sheet layup, after sintering and after fusing. The observations are set forth in the Table. 20

Table

<i>Process Step</i>	<i>Dry Blend of Invention</i>	<i>Dry Blend of Prior Art</i>
25 Sifting Dry Blend Prior to Sheet Layup	No filler separation from dry blend resin particles; powder was of uniform composition	Separation of filler and dry blend resin; the powder was no longer uniform in composition
30 Sheet Layup	Screeding roll remained free of filler and resin particle buildup	Screeding roll became covered with resin and filler particles
35 Sintering	Oven dwell time to desired degree of sinter: 2 Minutes at 375°F (190°C)	Oven dwell time to same degree of sinter as invention: 2.4 Minutes at 375°F (190°C) (20% increase in dwell time)
40 Resulting Fused, Consolidated Sheet	Glossy, smooth Surface	Surface less glossy, and less smooth than dry blend of invention

The above results indicate the improved processability obtained using the improved dry blend formulation of this invention when employed to produce floor coverings.

Examples 3 to 6

45 Using substantially the same procedure and materials of Example 1, four more floor coverings were produced. Each floor covering was separately formulated from a dry blend formulation employing 1 part of a different surfactant. The four surfactants employed were: Union Carbide L-7607, "Fluorad" FC430, "Zonyl" FSN and "Zonyl" FSC. Each formulation was observed during processing and found to exhibit improvements in processability similar to those of Example 1, as compared to the prior art. 45

Claims

50 1. A method of preparing a vinyl chloride dry blend from solid vinyl chloride dry blend grade resin particles, solid filler particles and liquid plasticizer, wherein the solid particles are blended, followed by the addition of the liquid plasticizer with heating to at least the fluff temperature of the solid resin particles such that the liquid plasticizer is uniformly and rapidly absorbed through to the core of each resin particle producing a dry powder of uniform composition, wherein there is mixed and added together with the plasticizer at least 0.2 part per 100 parts by weight of resin of a liquid cationic, nonionic, or amphoteric hydrocarbon-, silicone- or fluorocarbon-containing surfactant. 55

2. A method as claimed in claim 1 in which the surfactant is an alkylaryl polyether alcohol.

3. A method as claimed in claim 2 in which the alkylaryl polyether alcohol is octylphenoxy polyethoxyl ethanol.
4. A method as claimed in claim 1 in which the surfactant is an organo-modified silicone.
5. A method as claimed in claim 1 in which the surfactant is a nonionic fluorinated alkyl ester.
- 5 6. A method as claimed in any one of claims 1 to 5 in which the surfactant is employed in an amount within the range of from 0.2 to 2 parts per 100 parts by weight of resin. 5
7. The method of claim 1, carried out substantially as described in any one of Examples 1 or 3 to 6 herein.
8. A dry blend, whenever prepared by a method as claimed in any one of claims 1 to 7.
- 10 9. A dry blend comprising plasticized vinyl chloride resin particles, and filler particles, the composition comprising at least 0.2 per cent by weight, based on the weight of the unplasticized resin of a cationic, nonionic or amphoteric hydrocarbon-, fluorocarbon- or silicone-containing surfactant. 10
10. A dry blend as claimed in claim 9, as specified in any one of claims 2 to 7.
11. A decorative surface covering produced by screeding a dry blend prepared according to any one of claims 1 to 7, or as claimed in any one of claims 8 to 10, to a uniform gauge layup, sintering the resulting dry blend layup to form a porous sheet, printing the resulting porous sheet with vinyl plastisol ink and simultaneously fusing and consolidating the printed, sintered sheet to produce a decorative surface covering.
- 15 12. A surface covering as claimed in claim 11, which is a floor covering. 15